The influence of artificial weathering and treatment with FE–DBD plasma in atmospheric conditions on wettability of wood surfaces

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Abstract

The treatment of wood surfaces with plasma in atmospheric conditions is a well–known and researched processing technique. In this study, we introduce a new approach of wood surface treatment using a floating electrode dielectric barrier discharge (FE–DBD) plasma. The main principle of this kind of plasma is that wood represents an object for charge storage and the potential of the electrodes is changing according to the surroundings in the moment of voltage supply from high voltage source. The appearance of the discharge electric fields was firstly simulated with computer software and later analysed in real conditions. Additionally, plasma was characterised by optical emission spectroscopy as elemental analysis of the discharge. The designed FE–DBD technique was applied to some extent artificially weathered common beech (*Fagus sylvatica* L.) and Norway spruce (*Picea abies* (L.) Karst.) wood surfaces, in order to their re–activation and improvement of their wettability by commercial water-based coating. The results showed that contact angles of the droplets of applied liquids and waterborne coating decreased with weathering time, as well as after performed plasma treatment process.

1 Introduction

In manufacturing operations, fundamental differences between wood as a natural polymer composite, and other engineering solids or liquids pose numerous important technical challenges. Here, the mechanical resistance to separation of the interface between wood and liquid system (i.e. adhesion) plays an important role [1].

Since fundamental surface properties of a substrate are often governed by a layer of molecular dimensions, it is possible to modify this near–surface region without affecting the desirable bulk properties of the material [2, 3].

For all types of materials, it is characteristic that the properties of their surfaces change with time. From a technological, as well as from a quality point of view, these changes are usually reflected in a negative sense. With longer exposure to weather, colour changes of a wood surface reflect degradation of the surface and thus also changes of other properties [4, 5]. Physical processing (e.g. drying) and weathering of freshly cut wooden surfaces change the surface chemistry of wood in the manner of higher presence of extractives, and therefore the promotion of its insusceptibility for sufficient interactions with applied liquids [6–8].

Weathering of wood as a process is described as irreversible changes of the appearance and properties of a material as the effect of a long–term impact of weather, meaning solar radiation, air and oxygen contained in it, changes in temperature and humidity, as summing no direct influence of biotic factors [9, 10]. Resistance of wood surface against weathering can be assessed either with a longer natural weathering or with a shorter accelerated artificial weathering process (AWP). In the manner of research and manufacturing, natural weathering is time consuming and has a questionable repeatability of the process [11].

Precise determination of wettability of wood with contact angle (CA) measurements is inherently difficult because of its chemical heterogeneity, surface roughness, porosity and hygroscopic nature [6, 12, 13].

Due to the exposure to weathering, surfaces of wood become less susceptible to the absorption of applied liquids (de–activated) and, in the case of their surface protection, to treatment with coatings. In order to activate and improve their wetting behaviour and adhesion, different physical, mechanical and chemical surface treatments are used for generating active functional groups on the surfaces, which is required by many industrial applications (i.e. gluing, printing, coating) [14]. Active functional groups exposed (i.e. hydroxyl groups) increase surface free energy, which cause the surface to become more hydrophilic.

An efficient, clean, and economic alternative to activate wood surfaces prior to application of a coating system is the process of surface treatment with gas discharges or plasmas, respectively [3, 12, 14, 15–19]. Plasmas can be created under various conditions. For practical use in the wood industry, the most suitable way to perform the process uses air at atmospheric pressure [19–21]. In general, treatment with plasma induces surface chemical and physical modifications [17]. In the plasma treatment (PT) process, the surface of the treated substrate is exposed to plasma reactive species, such as ions, electrons, excited atoms and molecules [18]. After PT in air, the incorporation of oxygen–containing groups (–C–O, O–C–O, and O–C=O) on a woods´ surface can be indicative [19].

The field of wooden-surface PT at atmospheric pressure is dominated by dielectric barrier discharge (DBD) plasmas due to the low thermal impact [21]. The floating-electrode dielectric barrier discharge (FE–DBD) system is based on a conventional DBD, driven by alternating current high voltage applied between two conductive electrodes: a powered high voltage electrode and the second active electrode, which is not grounded and remains at a floating potential. One or both of these are covered with a dielectric to prevent transition to arc [22]. A "floating electrode" (FE) presents an object with a relatively high dielectric constant and the required capacity for charge storage [23], which is disconnected not only from the high-voltage terminal of the power supply, but also from the ground [24]. Such system has been used many times for medicine purposes like tissue sterilization, blood coagulation [22, 23, 25] inactivation and growth reduction of cancer cells [26, 27], or inactivation of bacteria on hands [28]. In these cases, the second electrode can be the human or animal skin, a sample, or an organ. The specific electrical behaviour in all these examples is induced by high water contents. In physical mechanisms, charged species of plasma are identified as the major contributors to the desired effect. The uniformity of FE–DBD treatments can range from a rather non-uniform continuous wave discharge to a uniform nanosecond–pulsed plasma [22].

In this study, we examined the properties of control and weathered surfaces of Norway spruce (*Picea abies* (L.) Karst.) and common beech (*Fagus sylvatica* L.) wood. The utilization of FE–DBD plasmas in the field of wood surface processing is entirely new and provides advantages that are especially beneficial for reactivation of weathered wood.

2 Materials and methods

2.1 Materials

In this study, the samples of common beech and Norway spruce wood with dimensions of (70.0 × 30.0 × 3.2) mm, with radial orientation of wood fibres were used. Before the start of the experiments, the substrates were conditioned in a chamber with a temperature of 20 °C and relative humidity 65 % to reach the equilibrium moisture content (12.1 % ¸at spruce wood and 10.8 % at beech wood).

The artificial weathering process of the samples took place in an Atlas SUN-TEST XXL + chamber (Atlas Material Testing Technology, Mount Prospect, USA), according to the standard EN ISO 11341 (2004) [29] in the interior mode. The xenon–arc lamp light parameters, equipped with a 3 mm window glass filter, and conditions in the chamber during the AWP test were as follows: irradiance at 340 nm set to irradiation power 0.35 $W \cdot m^{-2}$, relative humidity 65 %, chamber temperature 35 °C, and temperature on a black panel 55 °C. The series of samples were exposed to weathering for 5, 10 and 24 hours. After completion of each weathering interval, the series was taken out of the chamber for coating CA analysis.

2.2 Construction and assembly of the device for plasma generation

The FE-DBD device was constructed in AutoCAD (Autodesk, Mill Valley, U.S.A) and SolidWorks (SolidWorks Corp., Waltham, U.S.A.) software (*Fig. 1*). An aluminium and acrylic glass (Poly[methyl 2–methylpropenoate], PMMA) casing encompasses electrodes and HV power supply. The parameters of supplied alternating high voltage are regulated via a separate control unit. The plasma is ignited between the surface of the treated moving workpiece and two brass electrodes (15 mm diameter), both covered by a tubular ceramic as dielectric barrier (Al_2O_3, thickness 2.5 mm). Depending on the substrate to be treated, the distance between the dielectrics with electrodes, as well as the distance between the latter and the surface of the workpiece can be set. A workpiece is placed on a stage moving below the plasma module with a defined speed (0.5 to 6 $mm \cdot s^{-1}$) (*Fig. 1a*). Additionally, the construction of the device allows for the introduction of an additional working gas between the electrodes or on the surface of the workpiece, respectively (*Fig. 1b*).

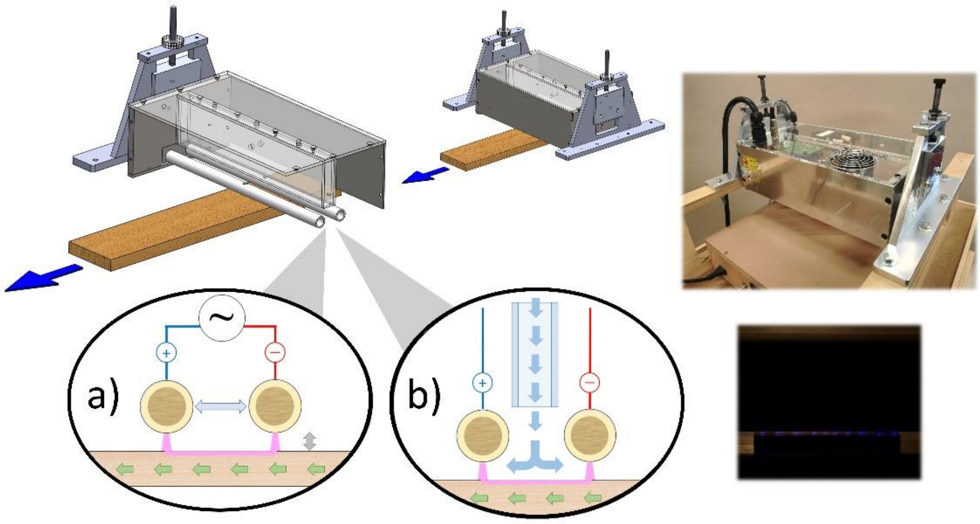

Fig. 1: Schematic presentation and picture of constructed device for treatment of wood surface with FE–DBD plasma: a) adjustable distance between the dielectrics with electrodes and adjustable the distance between the latter and the surface of the workpiece, b) the introduction of an additional working gas between the electrodes.

2.3 Computer simulation of the discharges

The distribution of the electric field for different configurations were simulated via the electrostatic module in COMSOL Multiphysics (COMSOL Inc., Stockholm, Sweden). Different setups and geometries were studied to predict the devices behaviour, i.e. the required peak voltages for plasma ignition and the likely localisation of the micro-discharges.

2.4 PT process of the wood surfaces

The surfaces of the individual sample was treated with a device with an FE–DBD (also known as direct Cold Atmospheric Plasma, dCAP) non–thermal plasma that generates plasma in air at atmospheric pressure. The photos of the discharges for the analysis of plasma appearance were taken by an Olympus E520 camera (Olympus, Tokyo, Japan), while the heat development during ongoing plasma treatments was studied by infrared camera Optris PI 160 (Optris GmbH, Berlin, Germany). An alternating high voltage (frequency 5 kHz, 20 kV peak voltage, peak current up to 90 A) from a pre–commercial power supply (HV power supply PG040C–0001 with control unit PG040B–0001, PlasmaGreen GmbH) was used to ignite the plasma at the surface of the workpiece at a moving rate of 3 mm $\cdot$ s^{-1}. In the experiments, the distance between the insulated electrodes was set to 5 mm, and the gap between the electrodes and the surface of the workpiece was approx. 1 mm.

During the PT of wooden samples the supplied electrical current and voltage were measured on the high voltage electrode by mixed signal oscilloscope Keysight Infinii Vision MSOX3024T (Keysight, Santa Rosa, U.S.A.), a fast passive high voltage probe with 100 MHz bandwidth, 1000 : 1 transmission ratio, and 40 kV maximum peak voltage (PHV4002–3–RO, dataTec AG, S/N 3978) and an inductive current clamp probe (N2893A, dataTec AG, S/N JP56102119).

2.5 Optical plasma diagnosis

A unique emission spectrum specific for the emitted photons and active species (molecules, atoms and ions) in discharge volume, generated in atmospheric conditions, which are important for plasma surface modification of wood [30–32] is identified with optical emission spectroscopy (OES). From these spectra, the temperatures of discharge species can be analysed [33]. Emission spectra of an atmospheric pressure air plasma with and without the wooden sample were measured with Avantes AvaSpec–3648 (Avantes BV, Apeldoorn, the Netherlands) optical spectrometer with a 3648–pixel CCD detector array and 75 cm focal length. The optical lens was placed 1 cm from generated discharge. Optical emission spectra were recorded with an integration time of 2 s and a resolution of 0.5 nm in the spectral range from 200 to 1100 nm.

2.6 Contact angle measurements

The applied droplets of the water-based commercial coating (Belinka Interier, Belinka Belles, d.o.o., Ljubljana, Slovenia) with a volume of 5 μL were applied on 6 different places of the radial surface on the each sample and followed by the Theta optical goniometer (Biolin Scientific Oy, Espoo, Finland). Apparent CA were measured by Young-Laplace analysis using the software (OneAttension version 2.4 [r4931], Biolin Scientific). All together 18 droplets for each type of sample were automatically analysed within 63 s (1.9 images per second). Measurement of the CA started when the droplet was separated from the dispenser, which occurred approximately 2 s after the first contact of the drop with the samples surface. In the case of PT samples, the measurements of CA were performed immediately to avoid effect of ageing.

3 Results and discussion

3.1 Computer simulations of electric fields

The appearance of the electric fields, given by COMSOL software calculations and simulations are shown in *Fig. 3*. The scales below the Figures are showing the absolute electric field values (in $V \cdot m^{-1}$). In order to compare different setups and geometries, the electrode potentials were set to +5 kV and –5 kV, respectively. The bottom border was set to ground potential, thus mimicking the moving stage as a grounded sample carrier. For electrode distances of 3 mm and below, a plasma ignition is to be expected only in–between the electrodes, thus allowing for a jet–type operation. At electrode distances of 5 mm and above, the device is operating most exclusively in a direct treatment mode of operation. Without substrate and for thin nonconductive substrates, the discharge resemble mostly a conventional direct DBD discharge between HV electrode and grounded stage. Further COMSOL studies (not shown) show that the influence of the stage become negligible at approx. 10 mm sample thickness, whereas the presented case still is within a transition area between the conventional DBD and the FE–DBD operation modes.

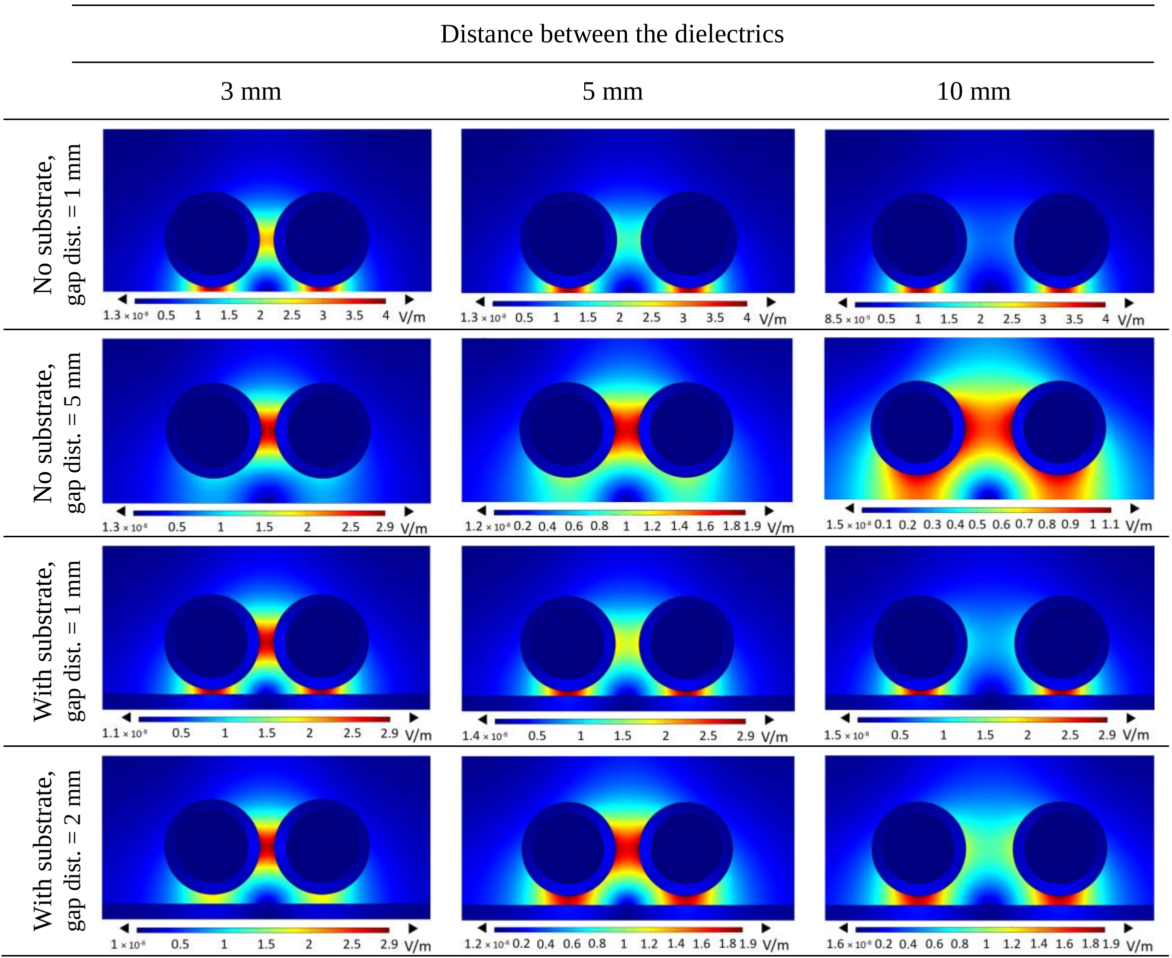

Fig. 3: Simulations of the strengths of electric fields ($\times 10^{-6}$ V $\cdot$ m^{-1}) by different gap distances, distances between the dielectrics and presence of the substrate, gained by COMSOL software.

In the next step, the results obtained by these simulations electric field are compared to the observed plasma conditions.

3.2 Plasma appearance

The photos and infrared imaging of the discharges, at different setups and geometries are shown in *Fig. 4*. At 3 mm distance between the dielectrics, plasma partially ignited both in–between the electrodes and in the discharge gap. By increasing the gap distance between the dielectrics (to 5 and 10 mm), the discharge appeared only in the gap below them, confirming the appearance of FE–DBD plasma. The temperatures of generated discharges were only about 2 °C higher than the temperatures of the surroundings, confirming the generated plasma as non–thermal.

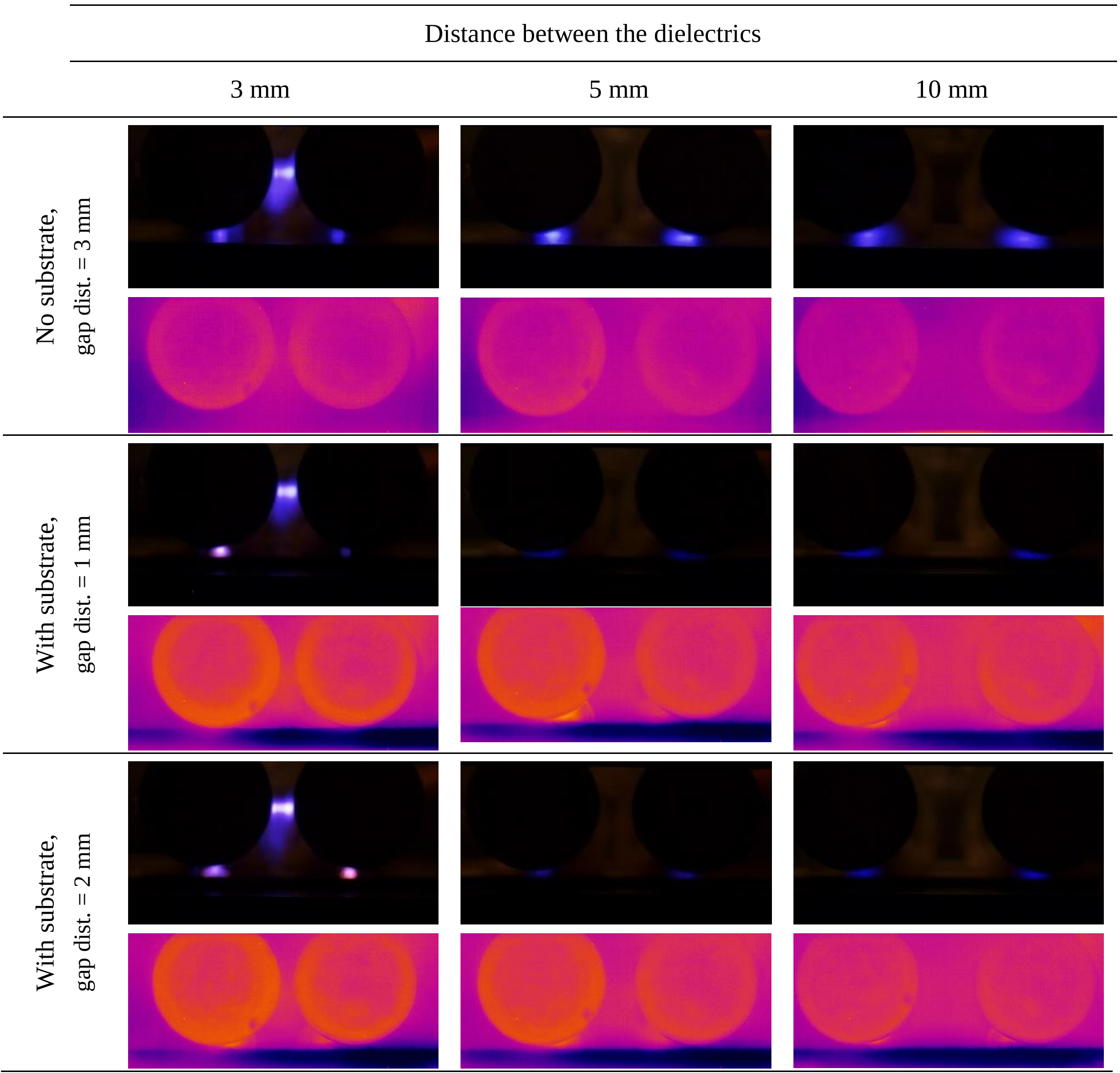

Fig. 4: The appearance of the discharges and thermal conditions (IR photos) by different gap distances, distances between the dielectrics and presence of the substrate.

3.3 Optical Emission Spectroscopy

Fig. 5 shows the OES spectrum from the FE–DBD operated by different voltages measured on high voltage electrode, generated in atmospheric conditions without wooden sample. The reactor–dependent pulse width amounts to approx. 25 µs for all setups used in this study. On the measured OES spectra, the most intense second positive system of N^2 emission lines between 290 and 450 nm could be identified (N_2 – 316 nm, 337 nm, 357 nm, 376 nm, 381 nm). Also a small N_2^+ emission line can be identified at 391 nm and at around 309 nm a small emission of OH radicals which is typical for humid air, originating i.a. from the oxidation of H_2O towards H_2O_2 and the subsequent decomposition into OH [32, 34–38].

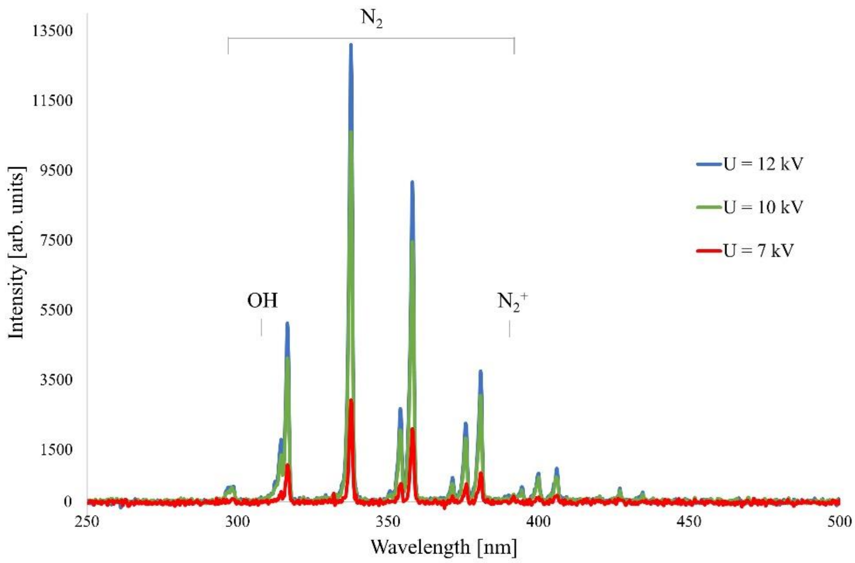

Fig. 5: OES spectra of atmospheric pressure air plasma at various voltages on the high voltage electrode (integration time 2 s).

After insertion of beech wood sample into discharge, no additional spectral features were identified, only the intensity of the most significant peaks proportionately decreased.

3.4 Coating contact angles

The CA of coating droplets, deposited on control and weathered surfaces of both types of wood species trivially differ between the shorter time (5 and 10 hours) of weathering. The exception were the surfaces weathered for 24 hours, where the CA were slightly higher. Additional PT of control and weathered samples surfaces of both woods caused the decrease of coating CA (*Fig. 6, Tab. 1*), meaning enhanced wettability of surfaces by the coating and its faster penetration into substrates.

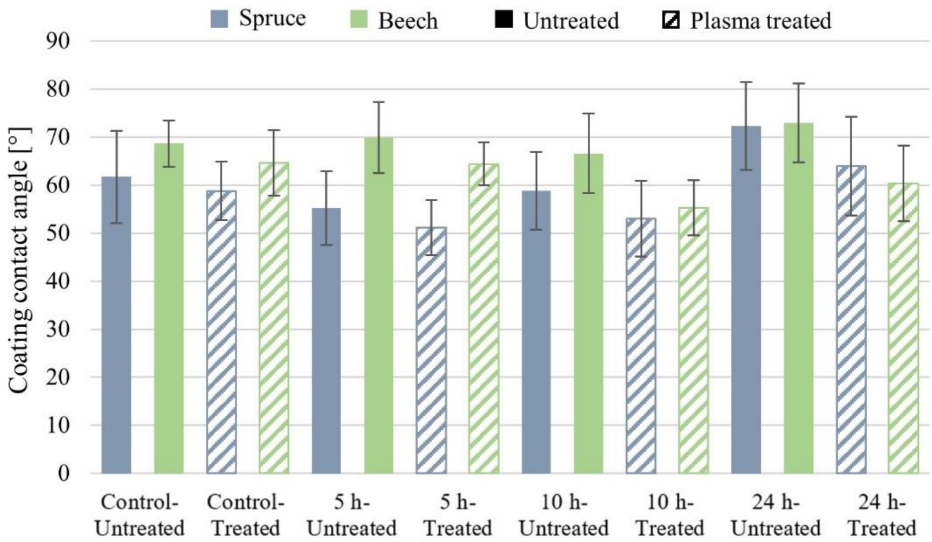

Fig. 6: Coating CA deposited on control and weathered samples surfaces.

Tab. 1: Coating CA deposited on control and weathered samples surfaces (statistics).

Wood type	Weathering time, surface type, coating contact angle (°)							
	Control-Untreated	**Control-Treated**	**5 h-Untreated**	**5 h-Treated**	**10 h-Untreated**	**10 h-Treated**	**24 h-Untreated**	**24 h-Treated**
Norway spruce	61.74 (9.63)	58.80 (6.10)	55.20 (7.66)	51.11 (5.73)	58.83 (8.07)	53.00 (7.90)	72.25 (9.14)	63.94 (10.30)
Beech	68.66 (4.77)	64.68 (6.80)	69.93 (7.37)	64.41 (4.50)	66.61 (8.26)	55.30 (5.80)	72.95 (8.21)	60.33 (7.89)

* The standard deviation is shown in parentheses.

4 Conclusion

Wettability and compatibility of wood surfaces with coatings can be improved via plasma treatments for both, fresh wood surfaces and wood surfaces after exposure to weathering. In this manner a new technique of dielectric barrier discharge plasma with a floating electrode, generated at atmospheric conditions, was introduced in the present paper. Computer software simulations of generated electric fields during plasma ignition showed their high dependence on configuration of the plasma reactor. These dependences were confirmed by plasma discharge observations in real conditions. Based on these results we can conclude that the treatment of wood surfaces is possible with newly introduced FE–DBD type of plasma in the air at atmospheric pressure. Finally, it was confirmed that this novel kind of PT technique can be used for re-activation of artificially weathered wood surfaces in mild conditions, contributing to better wettability of water-borne surface coatings.

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